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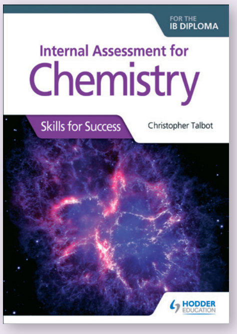

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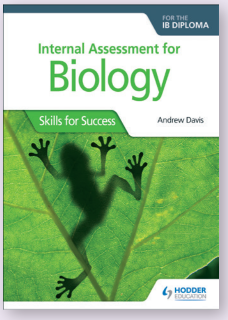

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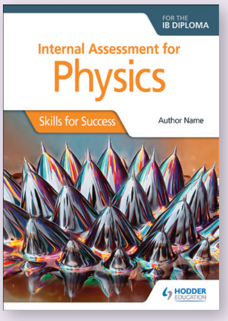

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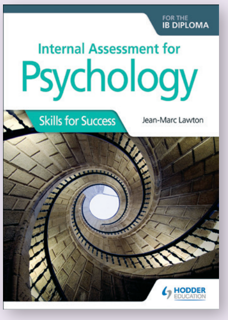

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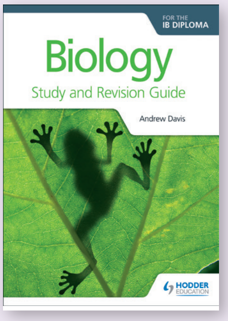

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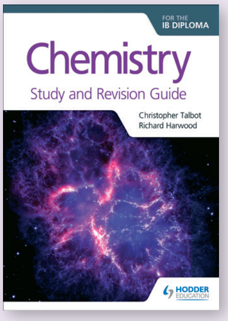

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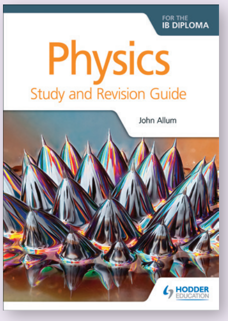

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Contents

2 Chemical analysis

Volumetric analysis

Standard solutions

- If there are impurities in the primary standard then the true mass present will be less than the measured mass. The solution will have a concentration less than the calculated value (a **systematic error** (Chapter 5)).
- The standard solution must be stable in air and in aqueous solution, or it will react with oxygen or carbon dioxide in the air or with water (hydrolysis).
- The standard solution must be soluble in water so that solutions of high concentrations can be prepared.
- The standard solution should have a large molar mass in order to minimize the percentage random **uncertainty** (Chapter 5) in the mass of substance weighed out.

Key definition

Standard solution – a solution with an accurately known concentration, prepared from a primary standard. This is a substance dissolved in a known volume of water to give a standard solution.

Some examples of reagents used as primary standards are outlined in Table 2.1.

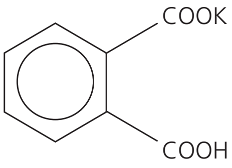
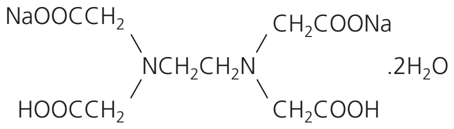
Primary standard	Examples
Acid	Hydrated ethanedioc acid, $(COOH)_2.2H_2O$ and potassium hydrogenphthalate: 
Base	Anhydrous sodium carbonate, Na_2CO_3
Oxidizing agent	Potassium dichromate(VI), $K_2Cr_2O_7$; potassium iodate(V), KIO_3
Reducing agent	Sodium ethanedioate, $(COONa)_2$
Complexing agent	Hydrated disodium salt of EDTA:  $(NaOOCCH_2)(HOOCCH_2)NCH_2CH_2N(CH_2COONa)(CH_2COOH).2H_2O$

Table 2.1 Common primary standards

ACTIVITY

1 Research the chemical properties of sodium hydroxide and explain why it is not a primary standard.

You need to calculate the mass of the primary standard required from the volume and concentration of solution required. The sample must be dried in a desiccator (Figure 2.1) to remove any water absorbed from the atmosphere. Primary standards can be dried by heating in an oven, but they may decompose at high temperature.

An empty dry weighing bottle is used to weigh out the primary standard, either by difference or by taring (see Chapter 1). The sample of the primary standard is then transferred to a beaker of distilled water and stirred with a glass rod.

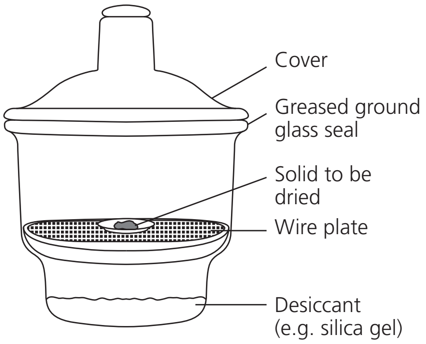


Figure 2.1 A laboratory desiccator

Expert tip

The stopper must be removed from the weighing bottle only when necessary to reduce the possibility of it re-absorbing water vapour.

The solution is added to a volumetric flask using a filter funnel (Figure 2.2). The inside of the beaker should be washed with distilled water and the washings transferred. The washing process should be repeated to make sure that all the primary standard has been transferred.

Distilled water is then added to the volumetric flask until the solution is close to the graduation mark. After the funnel is removed, distilled water is added using a dropper until the bottom of the meniscus is level with the graduation mark.

Expert tip

The volumetric flask should then be stoppered and turned upside down several times to ensure the solution is thoroughly mixed and is of uniform concentration (homogeneous).

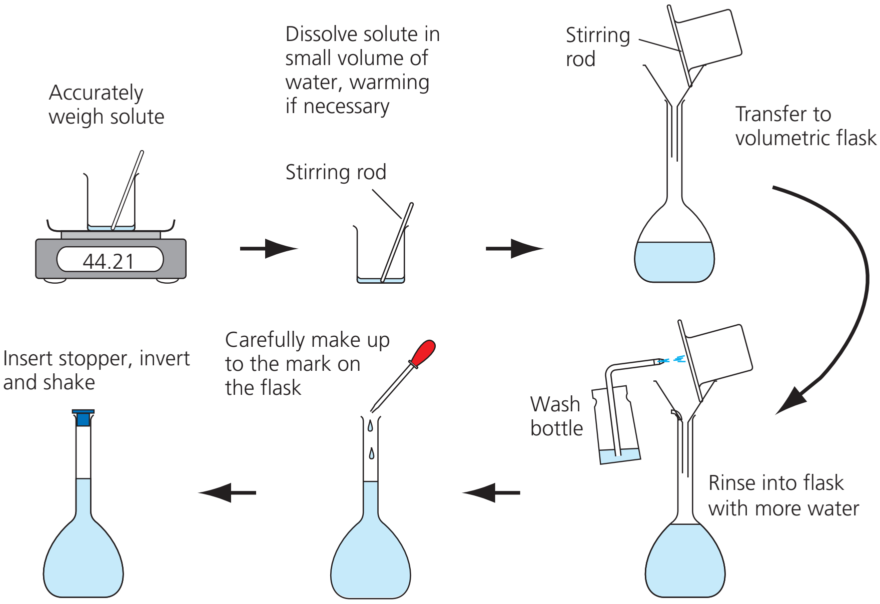


Figure 2.2 Preparing a standard solution

Titrations

A clean burette is rinsed with a small volume of the standard solution. It is then tilted to an almost horizontal position and rotated so the solution 'wets' the inside. The tip is rinsed by draining the solution through it. The burette is then clamped vertically and filled with the standard solution slightly beyond the zero mark; the tip is filled by opening the tap.

ACTIVITY

2 Explain the effect on an acid–base titration if the conical flask with the alkali is wet with distilled water before the acid solution is added to it.

Key definition

Titrant – the substance added from the titration.

With a white piece of paper behind the burette and with your eye level with the top of the standard solution, read the burette from the bottom of the meniscus and record the reading (Figure 2.3).

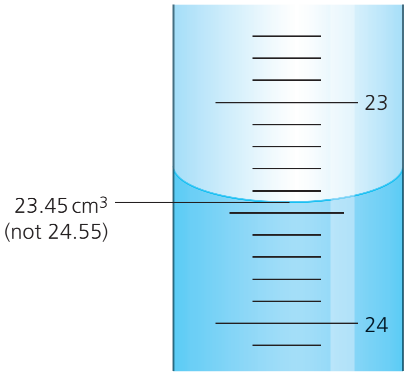


Figure 2.3 Recording a titre volume from a burette

Expert tip

When reading a burette, it is important to first remove the filter funnel you used to fill it. If this is left in place, some drops of solution could drain from it during the titration, leading to a false titre volume.

Use a pipette to transfer a fixed volume, usually 25.00 cm^3, of solution of unknown concentration (the analyte) to a conical flask. Clean the pipette by sucking up the analyte solution and wetting the surface inside by tilting and rotating it. The 'rinse' solution drains through the pipette tip and is disposed of.

Fill the pipette with the analyte solution to above the graduation (scratch) mark. Allow the solution to drain slowly from the vertical pipette until the bottom of the meniscus is level with the graduation mark.

Place the pipette tip in the neck of the conical flask and allow the analyte solution to drain. After it stops flowing, touch the tip against the inside wall of the flask for 30 seconds. Then add a few drops of an indicator to the analyte solution in the conical flask.

Place the conical flask containing the analyte solution and indicator under the burette, making sure that the tip of the burette is inside the neck of the conical flask. A white ceramic tile underneath the flask will allow you to see the colour change at the end-point more clearly (Figure 2.4).

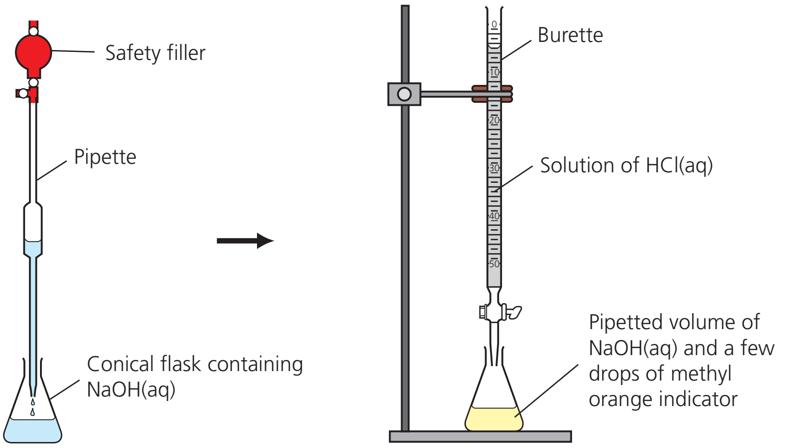


Figure 2.4 Apparatus for a titration

Examiner guidance

The first titration is a **trial run** to see the colour change of the indicator and to find an approximate value of the titre volume. Quickly add small volumes of the standard solution from the burette. You must swirl the conical flask to help the mixing process and give the reacting species in solution time to react. Continue doing this until the end-point is reached and then record the final burette reading.

Complete the titration by adding the standard solution very slowly, drop-wise, while swirling the conical flask. When the indicator just changes colour, you have reached the end-point of the titration. Record the final burette reading to 2 decimal places: the last number will be a 0 or 5.

The titre results of one person performing the same titration (or an other experiment) many times or of many students repeating the same titration should fall into a normal or Gaussian distribution curve (Figure 2.5).

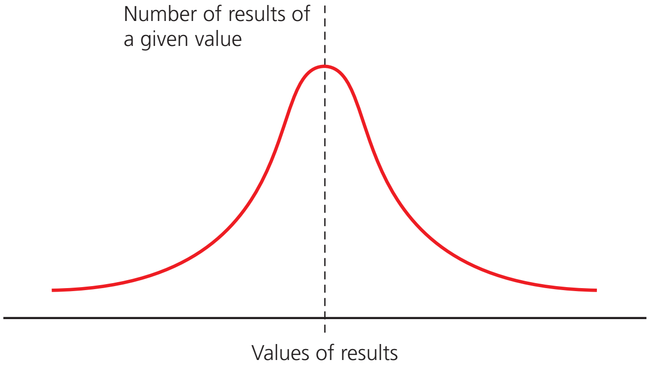


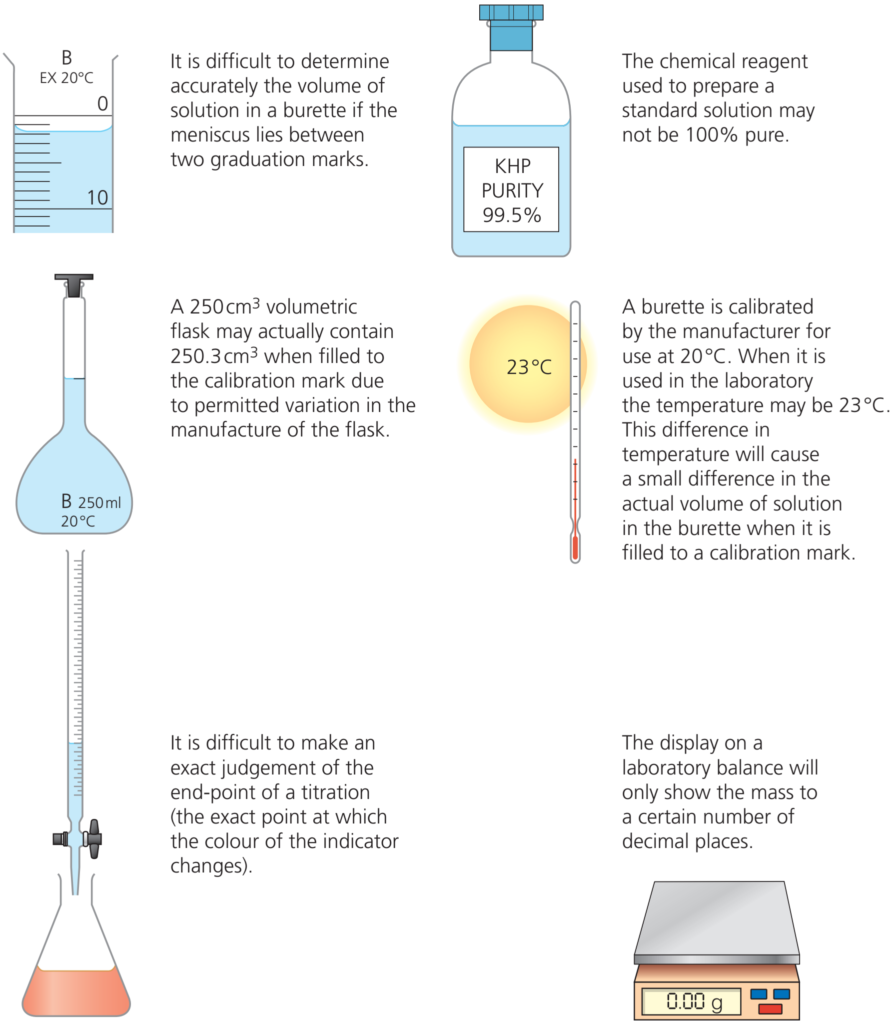
Figure 2.5 Normal or Gaussian distribution curve

Expert tip

If any of the solution splashes onto the walls of the flask then wash it into the mixture with distilled water. If you are close to the end-point and there is a drop of the solution stuck to the tip of the burette, remove it by touching the tip to the wall of the flask and washing it into the solution.

RESOURCES

http://www.rsc.org/Education/Teachers/Resources/practical/index3.htm



It is difficult to determine accurately the volume of solution in a burette if the meniscus lies between two graduation marks.

The chemical reagent used to prepare a standard solution may not be 100% pure.

A 250 cm^3 volumetric flask may actually contain 250.3 cm^3 when filled to the calibration mark due to permitted variation in the manufacture of the flask.

A burette is calibrated by the manufacturer for use at 20 °C. When it is used in the laboratory the temperature may be 23 °C. This difference in temperature will cause a small difference in the actual volume of solution in the burette when it is filled to a calibration mark.

It is difficult to make an exact judgement of the end-point of a titration (the exact point at which the colour of the indicator changes).

The display on a laboratory balance will only show the mass to a certain number of decimal places.

Figure 2.6 Sources of uncertainty in volumetric analysis

Expert tip

To minimize experimental errors (Figure 2.6) or increase reliability during titrations:

- it is important to swirl the mixture during titration as this ensures that the reactants are well mixed
- repeat your titration until you obtain at least two consistent results (two titre volumes that are within ±0.10 cm^3 of each other). Replication involves repeating the experiment a number of times
- always repeat the experiment to check your results are reliable. Calculate an average value to minimize random errors
- treat burettes with a cleaning mixture to remove grease, so they drain regularly
- prepare your sodium hydroxide solution freshly, since it absorbs carbon dioxide from the air which reduces its concentration.

Indicators

Choosing an indicator (Chapter 1) for a titration depends on the type of acid–base reaction taking place (Table 2.2).

Acid–base reaction type	pH at equivalence point	Suitable indicator
Strong acid/strong base	7	Any
Weak acid/strong base	>7	Phenolphthalein
Strong acid/weak base	<7	Methyl orange
Weak acid/weak base	~7	None

Table 2.2 Types of acid–base titration and choice of indicator

Key definition

Acid–base indicator – an indicator that changes colour on going from acidic to basic solutions.

Expert tip

It is important to use only a few drops (2 or 3) of indicator. This is because indicators are weak acids and so are neutralized by alkalis.

ACTIVITY

3 Find out about the double indicator method.

4 Distinguish between the terms **end-point** and **equivalence point**.

Types of titration

There are three main types of titration:

- **Acid–base titrations**, in which protons are transferred from the acid to the base.
- **Redox titrations**, in which an oxidizing agent is titrated against a reducing agent (or *vice versa*) usually in aqueous acidic solution. A redox titration may use a redox indicator that has one colour in its reduced state and a different colour in its oxidized state.
- **Complexometric titrations**, which are based on complex formation involving a Lewis acid–base reaction between metal ions and ligands, in which the ligands use their lone pairs of electrons to form coordinate bonds with metal ions.

The most common ligand (complexing agent) used is ethylenediaminetetraacetic acid (EDTA). It is a hexadentate ligand that forms very stable 1:1 complexes with metal ions (regardless of charge):

$$M^{n+} + EDTA^{4-} \rightarrow MEDTA^{n-4}$$

Most titrations are direct. 'Direct' means one reagent is added directly to the other until the end-point is reached. If a direct titration is not possible, a back titration may be used.

Back titrations

A back titration involves adding a known but excess amount of one standard reagent to a known mass of the substance being determined (the analyte). After the reaction between the two is complete, the excess amount of the standard reagent is determined by titration against a second solution of a primary standard.

Back titrations are used when:

- no suitable indicator is available for a direct titration
- the end-point of the back titration is clearer than that of the direct titration
- the reaction between the standard reagent and analyte is slow
- the analyte is insoluble in water.

Expert tip

A complexometric back titration is needed if:

- the metal ion precipitates in the absence of EDTA
- the metal ion reacts very slowly
- the metal ion forms an inert complex
- no suitable indicator is available.

In a complexometric back titration, a known excess of EDTA is added to the metal ion (buffered to an appropriate pH). Then the excess EDTA is titrated with a standard solution of a different metal ion.

Units of concentration

The SI unit for concentration is moles per cubic decimetre ($mol\,dm^{-3}$) but there are a number of alternatives for expressing the relative amounts of solute and solvent that you may encounter during practical work or in the chemical literature.

Molality

This is used to express the concentration of solute relative to the mass of the solvent. It has SI units of $mol\,kg^{-1}$. It is a temperature-independent measure of concentration and is used when the osmotic properties of the solution are relevant.

Percent composition (% w/w)

This is the solute mass (in g) per 100 g of solution. The advantage is that a solution can be prepared easily by pre-weighing the solvent and solute and then mixing.

Worked example

Calculate the concentration in % (w/w) for a 1.00 M solution of sodium chloride, NaCl(aq).

$$\text{Concentration} = \frac{1\,\text{mol}}{1\,\text{dm}^3} = \frac{58.44\,\text{g}}{1\,\text{dm}^3} = \frac{5.844\,\text{g}}{100\,\text{cm}^3} = 5.8\%(\text{w/w}).$$

Percent concentration (% w/v and % v/v)

This is the solute mass (in g) per 100 cm^3 of solution. This is more commonly used than percent composition, since solutions can be accurately prepared by weighing out the required mass of solute and then making this up to a known volume using a volumetric flask. The equivalent expression for liquid solutes is % v/v.

Parts per million (ppm)

This is a non-SI weight per volume (w/v) concentration term commonly used to describe very low concentrations of chemicals. The term ppm is equivalent to the expression $\mu g\,cm^{-3}$ ($10^{-6}\,g\,cm^{-3}$) and a 1.0 ppm solution of a substance will have a concentration of 1.0 $\mu g\,cm^{-3}$.

ACTIVITY

5 250 cm^3 of water contains 2.2 mg of dissolved oxygen. Determine the concentration in ppm.

6 The molarity of white vinegar ($CH_3COOH(aq)$) is 0.8393 $mol\,dm^{-3}$. The density of white vinegar is 1.006 $g\,cm^{-3}$. The molar mass of ethanoic acid is 60.06 $g\,mol^{-1}$. Calculate the mass percent composition (w/w) of white vinegar.

7 Find out about the use of 'volume strength' as a measure of the concentration of hydrogen peroxide.

Redox titrations with starch and iodine

The starch solution is added to the reagent in the flask at the start of the titration and the end-point is from colourless to blue. If the iodine solution is being titrated, the starch must be added later because the concentration of iodine would be so high that some of the molecules would interact permanently with the starch and not be free to react with the titrant.

The starch is therefore added once most of the iodine molecules have been reduced; that is, when the initial brown colour of the solution has faded to a very pale yellow colour. On adding the starch, the solution turns blue. The titration is complete when the blue colour disappears.

Expert tip

A freshly prepared starch solution must be used in iodine titrations. It is photosensitive and decomposes quite rapidly to form glucose which is a mild reducing agent and will react with the iodine, causing a systematic error in the titre volume.

ACTIVITY

Some iron tablets, containing iron(II) sulfate, were left open on the shelf so that some of the iron(II) sulfate was oxidized into iron(III) sulfate.

You are to analyse by titration the percentage by mass of iron(II) ions present. The partially oxidized iron tablets are dissolved in water to release the iron(II) and iron(III) ions. A suitable volume of dilute sulfuric acid is then added to the solution to prevent oxidation of iron(II) to iron(III) ions.

An unoxidized sample of an iron tablet is dissolved and made up to 250.00 cm^3. 25.00 cm^3 of the iron(II) ions in solution required 14.00 cm^3 of potassium dichromate(VI) to reach an end-point in a titration.

8 Calculate the mass of iron(II) sulfate in the unoxidized sample of iron tablet that was used.

Ionic reaction:

$$6Fe^{2+} + Cr_2O_7^{2-} + 14H^+ \rightarrow 6Fe^{3+} + 2Cr^{3+} + 7H_2O$$

9 Outline how the iron(III) ions present in the solution formed from a dissolved iron tablet can be converted to iron(II) ions and then analysed by a redox titration.

10 Outline how that procedure could be modified, using powdered pure zinc, to determine the amount of iron(III) ions in an oxidized iron tablet sample.

Ideas for investigations involving titration

- Analyse wines (dichromate(VI)) or iodine and starch.
- Investigate nitrification in filtered soil solution via back titration.
- Analyse the vitamin C content of fruit juices (using DCPIP, iodine or iodate(V) ions).
- Investigate the titration of decarbonated fizzy drinks using sodium hydroxide and phenolphthalein.
- Determine the chloride ions in cheese using the Volhard method.
- Investigate calcium and magnesium ion concentration (using EDTA in the presence of eriochrome black T indicator).
- Investigate the copper and zinc content of brass using redox titrations.
- Investigate purity and composition of antacids by back titration.
- Determine the concentration of bleach (sodium chlorate(I) solution) by redox titration
- To investigate the reaction between bromine and thiosulfate ions via a redox titration.

Gravimetric analysis

Gravimetric analysis involves the accurate measurement of the mass of a product from an accurately measured mass of a reactant. There are two types: volatilization and precipitation.

In a volatilization method, you weigh out a sample of the analyte and heat it to constant mass using a crucible (Figure 2.7). You can then collect the volatile product and weigh it, or determine its mass indirectly from its loss in mass. You can use a desiccator (Chapter 1) to store the sample while it is cooling down.

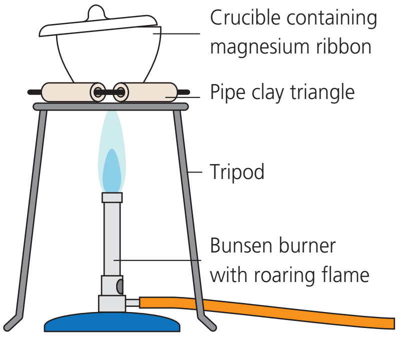


Figure 2.7 Gravimetric analysis using a crucible to heat magnesium to constant mass

In a precipitation method, you dissolve the analyte in water and convert it into an insoluble product by reacting it with another chemical (reagent). Then filter, wash, dry and weigh the precipitate.

In precipitation methods, the particle size of the solids should be large because large particles are easier to filter. Small particles could block the filter paper or pass through it. A solid that consists of large particles has a smaller total surface area and so can be washed free of impurities more easily.

Stir rapidly and heat the reaction mixture on a steam bath.

Expert tip

To minimize experimental errors or increase reliability for gravimetric analysis:

- heat the solid evenly to make sure all of the solid has undergone decomposition
- cool the heated sample in a desiccator to prevent the absorption of water vapour from the air
- repeat the heating–cooling–weighing cycle until constant mass occurs
- ensure all the ions are precipitated out during a precipitation gravimetric analysis by using excess precipitating reagent
- wash the filtered residue with a suitable solvent that will remove impurities, but does not dissolve the residue
- plan to have sufficient precipitate so the effect of random uncertainty in the measurements is relatively low.

Key definition

Precipitation reaction – a reaction that involves the formation of an insoluble salt when two solutions containing soluble salts are combined. The insoluble salt formed is known as the **precipitate**.

ACTIVITY

11 Magnesium sulfate does not decompose when heated strongly with a Bunsen burner. However, magnesium carbonate decomposes on heating to form magnesium oxide and carbon dioxide.

You are to design an experiment to determine the percentage by mass of magnesium carbonate in a sample of magnesium carbonate contaminated by magnesium sulfate.

Outline the practical sequence for the method to record appropriate masses, decompose magnesium carbonate in the sample by heating and ensure that thermal decomposition is complete. Show how you would tabulate your results.

Ideas for investigations involving gravimetric analysis

- Determine chloride ion concentration in sea water or foods (using silver nitrate solution).
- Analyse water of crystallization in hydrated salts.
- Analyse mixtures of two salts; analyse impure sodium thiosulfate.
- Investigate the reaction between copper(II) sulfate and excess sodium carbonate solution.

Colorimetric and spectrophotometric analysis

Colorimetric analysis is used to determine the concentration of analytes that are coloured or can be converted quantitatively by a chemical reaction into a coloured species.

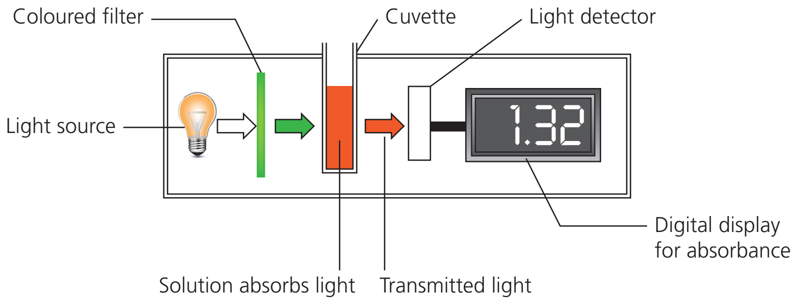


Figure 2.8 The pathway of visible light in a colorimeter (a blue or green filter is used with a red solution)

The colour of a solution depends on the frequencies/wavelengths of light it absorbs (and this in turn determines the wavelengths of light that are transmitted). The intensity of its colour depends on the concentration of the solution: the more concentrated the solution, the darker its colour, as it absorbs more light.

Examiner guidance

Generally speaking, there is a linear relationship between the concentration of a solution and the absorbance (known as the **Beer–Lambert law**. However, high absorbance values above 2 will generally not obey this linear relationship. If readings of over 2 are obtained, dilute the sample by a known amount and re-measure the absorbance.

A standard calibration line often has to be obtained; this involves preparing a series of solutions of known concentration by the accurate dilution of a standard solution. The absorbance values of these standard solutions are measured using the instrument (with the correct filter or correct wavelength, for maximum absorption).

Expert tip

You will need to find out how to operate your school's UV-vis spectrophotometer or colorimeter (Figure 2.8) and be familiar with a colour wheel and the concept of complementary colours.

Expert tip

When you handle the cuvette you should have clean fingers. Two opposite faces of the cuvette (plastic or glass) are ribbed so when you place the cuvette in the instrument, you must ensure that the beam of light passes through the transparent flat faces. Otherwise the light will scatter and cause a large systematic error in your absorbance reading.

Key definition

Beer–Lambert law – the linear relationship between absorbance and oncentration of an absorbing species.

ACTIVITY

12 Plot a calibration line for the following data for aqueous solutions of chromium(III) nitrate and use it (via **interpolation**) to calculate the concentration of a solution with an absorbance of 0.256.

Solution number	Concentration/mol dm^{-3}	Absorbance
1	0.020	0.333
2	0.010	0.163
3	0.005	0.084
4	0.0025	0.041
5	0.0013	0.019
6	0.00065	0.011

Ideas for investigations involving colorimetric or spectrophotometric analysis

- Investigate the movement of coloured transition metal ions through a semi-permeable membrane.
- Determine the concentration of lactose in milk via colorimetry using bicinchoninic acid.
- Determine the formula of a hydrated transition metal ion complex.
- Investigate the extraction of iron or manganese from tea by oxidation of cations followed by colorimetric analysis.
- Investigate the reaction between copper(II) ions and aspirin via colorimetry in the presence of potassium hydrogencarbonate.

Many laboratory procedures, including titrations and spectrophotometry, may involve dilutions.

Dilutions

Key definition

Serial dilution (Figure 2.9) – any dilution where the concentration decreases by the same factor in each successive step.

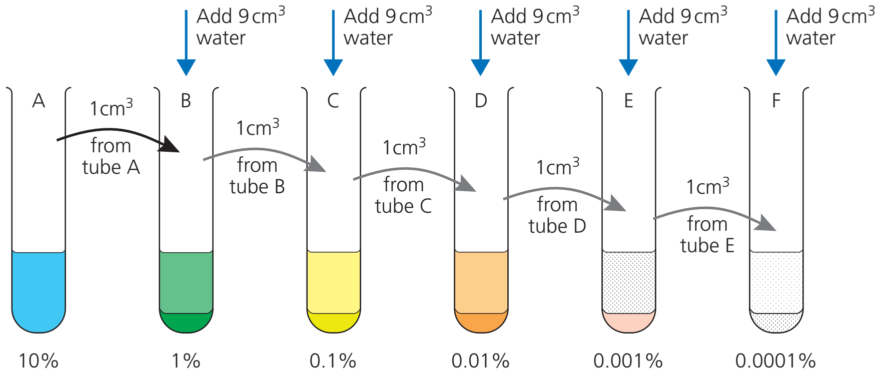


Figure 2.9 Making a serial dilution: dilution factor of 10 for each transfer

For example, a $\frac{1}{10}$ dilution of a solution represents 1 part of the chemical solution added to 9 parts of diluent (usually water). Serial dilutions are multiplicative. For example, if a $\frac{1}{8}$ dilution of the chemical stock solution is made followed by a $\frac{1}{6}$ dilution with water, then the final dilution is $\frac{1}{8} \times \frac{1}{6} = \frac{1}{48}$.

Doubling dilutions are a series of ½ dilutions: each successive solution will contain half the amount of the original concentrated (stock) solution. Table 2.3 shows the effect of doubling dilutions performed six times. This results in a series of dilution, each a doubling dilution of the previous one.

Dilution	Concentration as a fraction of the original
First dilution	$\frac{1}{2}$
Second dilution	$\frac{1}{2} \times \frac{1}{2} = \frac{1}{4}$
Third dilution	$\frac{1}{2} \times \frac{1}{2} \times \frac{1}{2} = \frac{1}{8}$
Fourth dilution	$\frac{1}{2} \times \frac{1}{2} \times \frac{1}{2} \times \frac{1}{2} = \frac{1}{16}$
Fifth dilution	$\frac{1}{2} \times \frac{1}{2} \times \frac{1}{2} \times \frac{1}{2} \times \frac{1}{2} = \frac{1}{32}$
Sixth dilution	$\frac{1}{2} \times \frac{1}{2} \times \frac{1}{2} \times \frac{1}{2} \times \frac{1}{2} \times \frac{1}{2} = \frac{1}{64}$

Table 2.3 Series of dilutions

The dilution factor is calculated from the formula: $\frac{\text{final volume}}{\text{aliquot volume}}$.

Worked example

Deduce the dilution factor if you add a 0.1 cm^3 aliquot of a specimen to 9.9 cm^3 of diluent.

The final volume is equal to the aliquot volume plus the diluent volume:

$$0.1\,cm^3 + 9.9\,cm^3 = 10\,cm^3$$

The dilution factor is equal to the final volume divided by the aliquot volume:

$$\frac{10\ cm^3}{0.1\ cm^3} = 1:100 \text{ dilution}$$

Chromatography

All types of chromatography involve a stationary phase (usually silica (SiO_2) or cellulose) and a mobile phase (usually a solvent system). Two compounds usually have different partitioning characteristics between the stationary and mobile phases. Since the mobile phase is moving, then the longer the time a compound spends in that phase, the further it will travel.

Analytical techniques may be used to follow the course of reactions and determine the purity of products. These methods include GC, HPLC and TLC. Sample sizes are usually from microgram to milligram quantities.

Preparative methods are used to purify and isolate organic compounds for characterization or further use. The common techniques are preparative HPLC, preparative TLC and column chromatography.

Ideas for investigations

Ideas for possible investigations involving paper chromatography include:

- investigating the presence of different chloroplast pigments
- studying pigments in red and green peppers
- studying hydrolysed proteins (using locating agents)
- studying inorganic ions and inorganic precipitates.

Possible investigations involving TLC include:

- monitoring the esterification of benzene carboxylic acid
- separating of phenols
- investigating the products of the nitration of phenol
- isolating organochlorine pesticide residues.

Expert tip

Sometimes a single solvent is used in preparative chromatography, but usually it is a binary mixture of solvents with different polarities. The advantage is that the bulk polarity can be controlled by varying the ratio of the two solvents.

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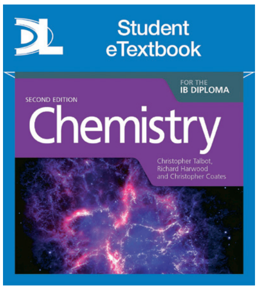

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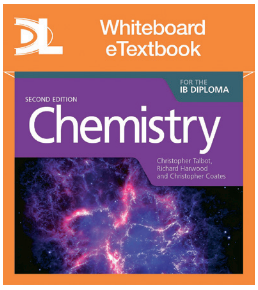

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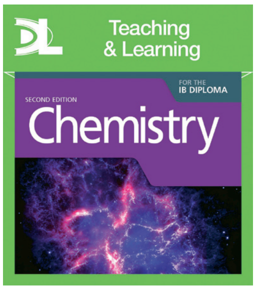

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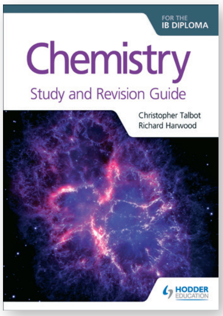

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